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Implementation Challenges for Sintered Silicon Carbide Fiber Bonded Ceramic Materials for High Temperature Applications

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Abstract

During the last decades, a number of fiber reinforced ceramic composites have been developed and tested for various aerospace and ground based applications. However, a number of challenges still remain slowing the wide scale implementation of these materials. In addition to continuous fiber reinforced composites, other innovative materials have been developed including the fibrous monoliths and sintered fiber bonded ceramics. The sintered silicon carbide fiber bonded ceramics have been fabricated by the hot pressing and sintering of silicon carbide fibers. However, in this system reliable property database as well as various issues related to thermomechanical performance, integration, and fabrication of large and complex shape components has yet to be addressed. In this presentation, thermomechanical properties of sintered silicon carbide fiber bonded ceramics (as fabricated and joined) will be presented. In addition, critical need for manufacturing and integration technologies in successful implementation of these materials will be discussed.

Implementation Challenges for Sintered Silicon Carbide Fiber Bonded Ceramic Materials for High Temperature Applications

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Outline

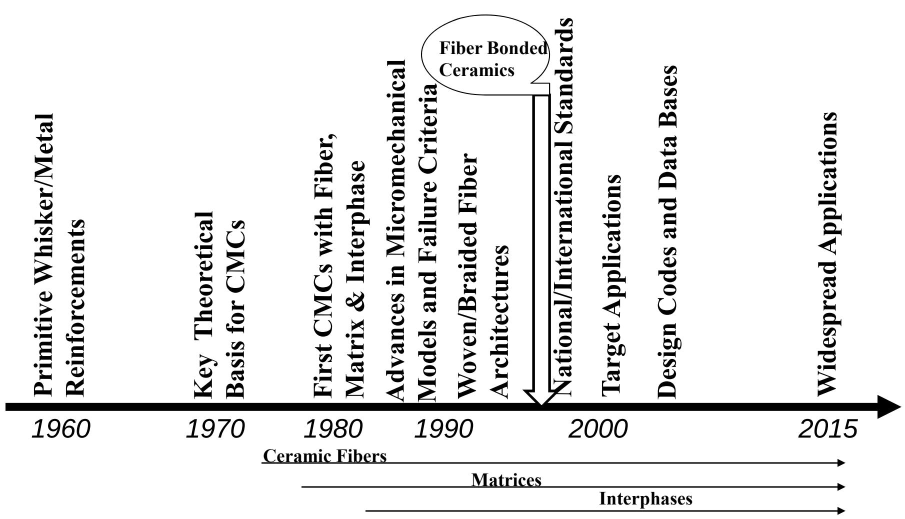
- **Introduction and Background**
- **Fiber Bonded Ceramics: *Overview***
  - ***Materials and Manufacturing***
  - ***Microstructure (SEM, TEM)***
  - ***Thermal Properties***
- **Key Implementation Challenges**
  - ***Thermomechanical Performance***
  - ***Integration Technologies***
  - ***Robust Manufacturing and Cost***
- **Concluding Remarks**
- **Acknowledgments**

Ceramic Matrix Composites (CMCs): *Past, Present and Future ?*

- **Tremendous potential for use of ceramic matrix composites in aerospace and ground-based applications.**
- **Many intrinsic advantages over other material classes.**
- **Unique capabilities relative to certain applications.**
- **Substantial, long-term government funding for research and development in these materials worldwide.**
- **But many scientific, technical, economic, and cultural problems *still remain* in wide scale use of these materials.**

Introduction/Background

Timescale for Development and Applications of CMCs



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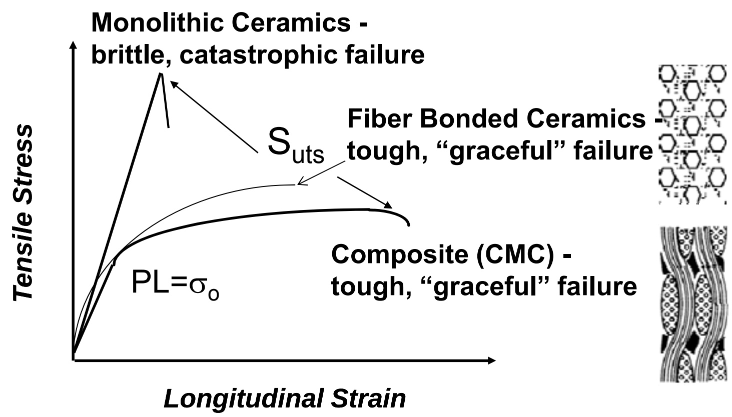
Introduction/Background

Although more complex and more expensive than monolithic ceramics, the added “toughness” of CMCs make them more inherently damage tolerant than monolithic ceramics.
(Fiber Bonded Ceramics Could Play a Key Role)



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SiC Fiber Bonded Ceramics

Processing and Microstructure

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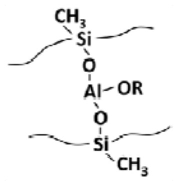
Synthesis of Amorphous Si-Al-C-O Fiber

Polyalumino-carbosilane



Melt spinning

@220°C

Forming continuously to fiber shape

Curing

in Air @160°C

Firing

in Ar gas up to 1300°C

Changing to ceramic fiber

Amorphous Si-Al-C-O fiber

Si	C	O	Al
53	33	13.3	0.7

T. Ishikawa, Ube Industries

Fabrication of SiC Fiber Bonded Ceramics (SA-Tyrannohex)

Amorphous Si-Al-C-O fiber

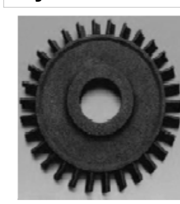
Fabric (8-harness satin)

Cut & Lamination

Hot Pressing

at ~1900°C, 50MPa, 1h in Ar gas

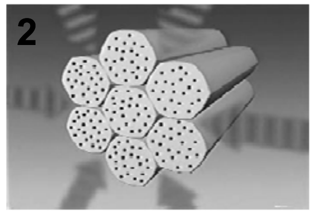
SA-Tyrannohex™



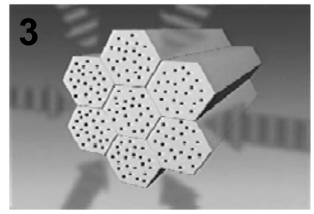
Ube Industries

Forming Process of SA-Tyrannohex during Hot Pressing

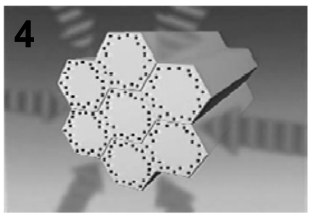
Under high pressure & high temp. in a hot press



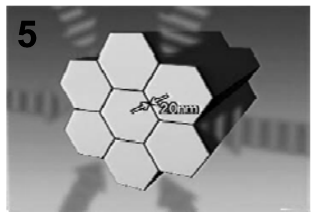
Deforming fibers & Evaporating SiO and CO gas



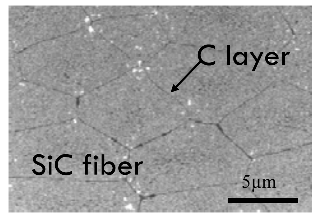
Closed-pack hexagonal columnar structure



Carbon diffusion from the center of fibers to its surface



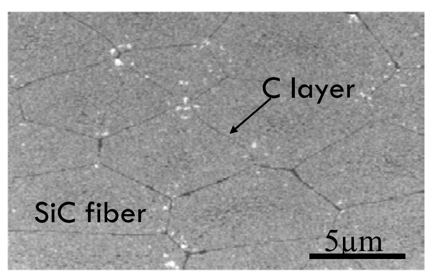
Unique SA-Tyrannohex structure

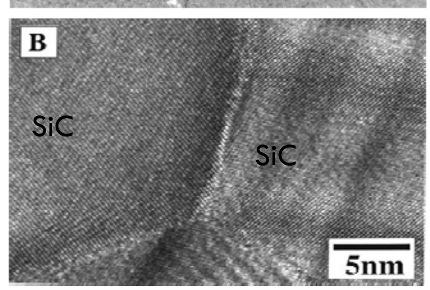


T. Ishikawa, Ube Industries

Microstructure of Fiber Bonded Ceramics

- ❑ **Unique Microstructure**
  - ❑ Hexagonal columnar SiC polycrystalline fibers
  - ❑ 10-20 nm turbostratic carbon interfacial layers
- ❑ **Good Properties**
  - ❑ High density (~98-99%)
  - ❑ High thermal conductivity (33 W/mK @ 1600°C)
  - ❑ High strength sustained up to 1600°C
    - ❑ Flexural strength~300 MPa
  - ❑ High fracture toughness (1200 J/m^2 @ RT)

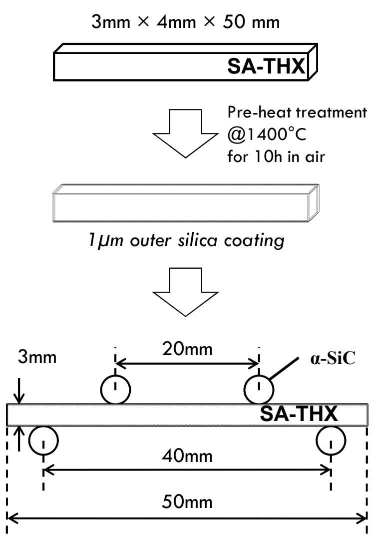


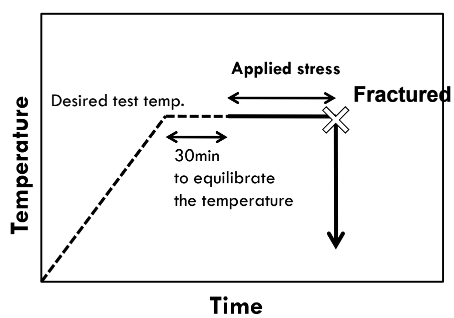


SiC Fiber Bonded Ceramics

Thermomechanical Properties

Thermomechanical Evaluation of Flexural Stress Rupture Behavior *(time-to-failure)*





- **Fractography of tested specimens in SEM**
  - Focused on the tensile side

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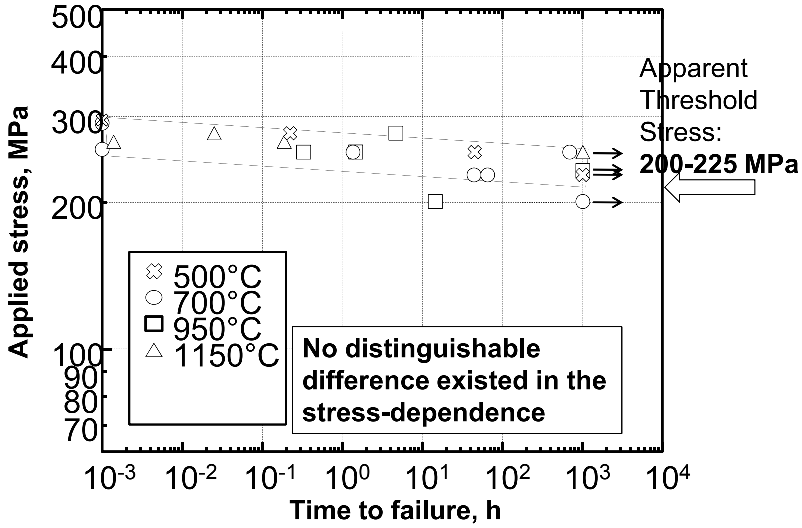
Stress versus Lifetime Behavior for SA-Tyrannohex™ Tested up to 1150 C in air



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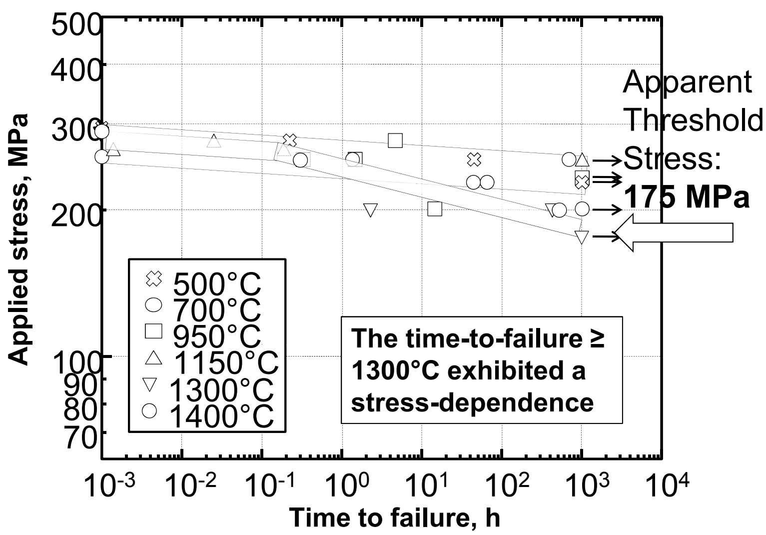
Stress versus Lifetime Behavior for SA-Tyrannohex™ Tested up to 1400° C in Air



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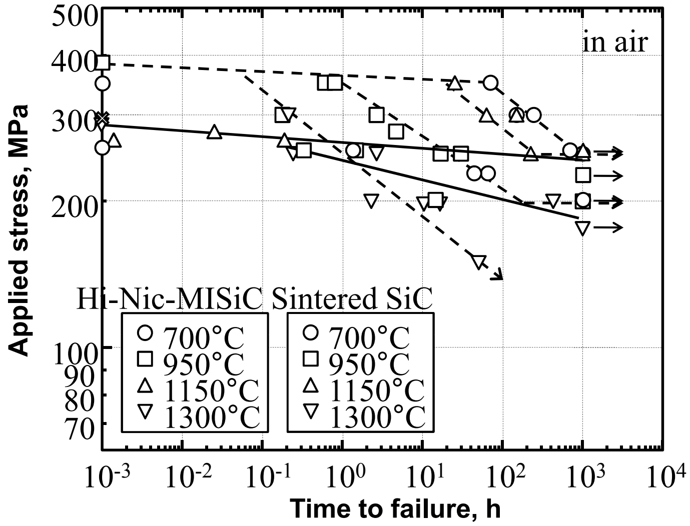
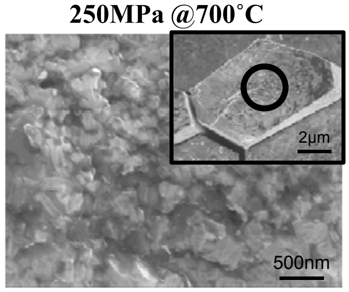
Stress versus Lifetime Behavior of MI SiC/SiC and SA-Tyrannohex up to 1300°C in Air

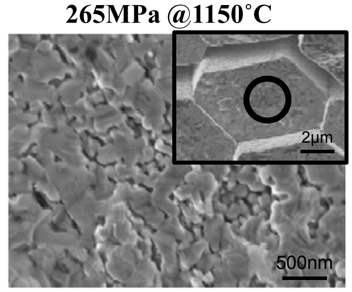


Fractography of SA-Tyrannohex after the Rupture Tests between 700-1400°C

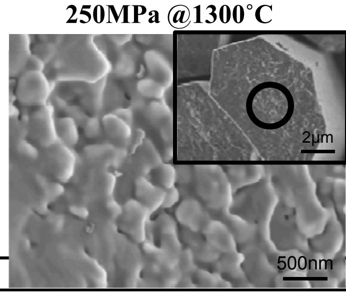
250MPa @700˚C



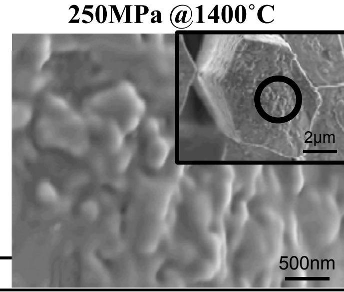
265MPa @1150˚C



250MPa @1300˚C



250MPa @1400˚C



SiC Fiber Bonded Ceramics

Joining and Integration Technologies

Critical Needs for Integration Technologies

- **A wide majority of CMC components have to be integrated with existing metallic constituents or components either during component manufacturing or in service.**
- **It is important to understand the technical issues among the different material systems**
- **Robust integration technologies can also play a key role in manufacturing of large size components (beyond existing manufacturing capabilities) utilizing building block approach.**
- **Building Block approach has been used through the ages and currently quite effectively in metal, polymer, and electronic industry.**

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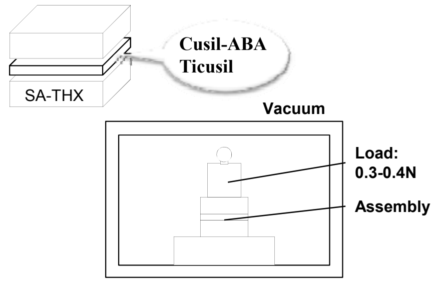
Active Metal Brazing of Fiber Bonded Ceramics

- Materials
  - SA-Tyrannohex with two orientations
    - Parallel type
    - Perpendicular type
  - AgCuTi brazing alloy
    - Cusil-ABA
    - Ticusil
- Conditions
  - Temperature: 10-15°C above liquidus
  - Hold time: 5 min
  - Vacuum level: 10^{-6}Torr

- Procedure



Name	Composition (wt%)	T_L (K)	T_S (K)	CTE ($\times 10^{-6} K^{-1}$)
Cusil-ABA	63.0Ag-35.25Cu-1.75Ti	1088	1053	18.5
Ticusil	68.8Ag-26.7Cu-4.5Ti	1173	1053	18.5

Characterization
- Mounted in epoxy, and polished
- Optical microscopy
- SEM-EDS

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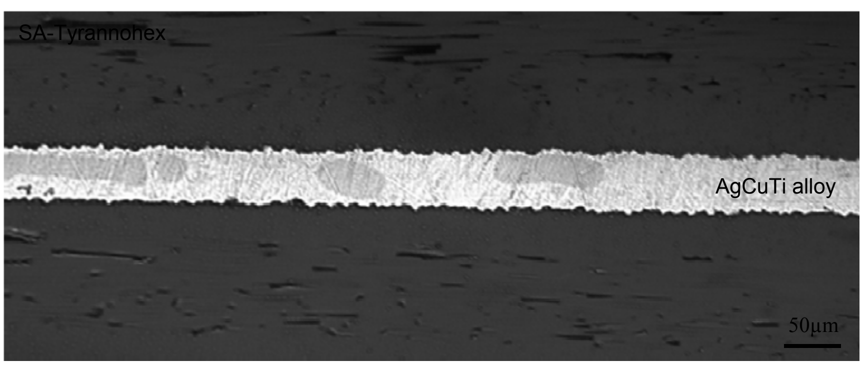
Brazing of SA-Tyrannohex Material to Itself using the AgCuTi Brazing Alloy



Uniform joint microstructure and good bonding

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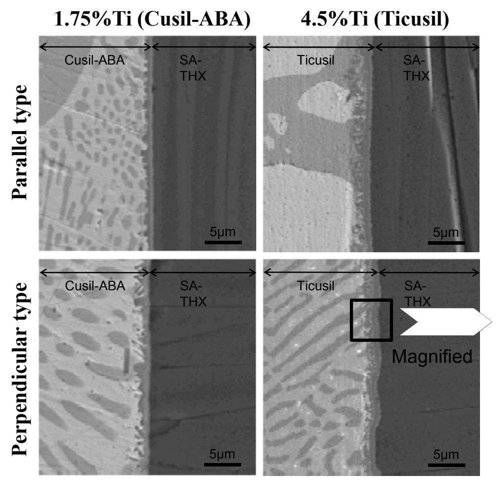
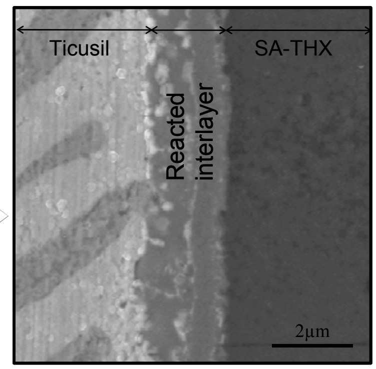
Joining of SA-Tyrannohex Material to Itself using the AgCuTi Brazing Alloy (Cusil-ABA & Ticusil)



Despite orientation of fiber and Ti content, microstructures of the joints were very similar



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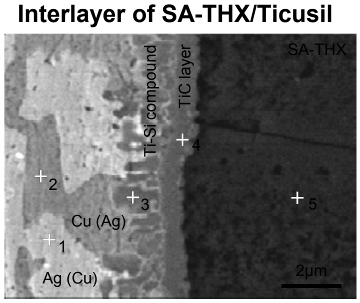
Microstructure of the Joint Interfaces

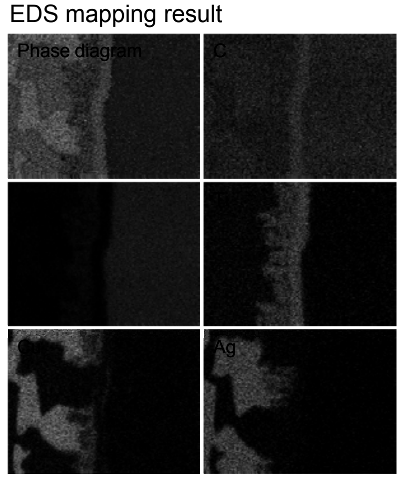
- **Interlayer of SA-THX/Ticusil**



atomic %

Pos.	Al	Si	Ti	Cu	Ag
1	0	0	0	16.0	84.0
2	0	0	0	98.5	1.5
3	0	39.0	50.1	10.9	0
4	0	8.0	87.9	4.1	0
5	1.1	98.5	0	0	0

- EDS mapping result



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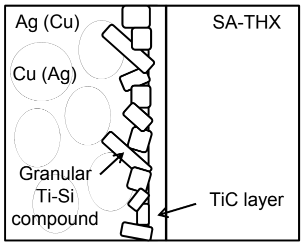
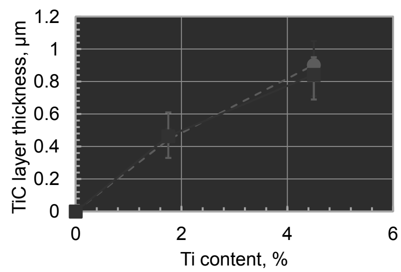
Summary of Joint Microstructure of SA-THX Materials using AgCuTi Alloy Brazes

- **Schematic**



- **In the interface between SA-Tyrannohex and the metals, thin TiC layer and granular Ti-Si compound (could be Ti_5Si_3) were formed regardless of the orientation and Ti content.**
- **The joint microstructure of the SA-Tyrannohex using Ag-Cu-Ti alloy brazes was very similar regardless of the orientation and Ti content.**

- **Effect of Ti on the thickness of TiC layer on SA-Tyrannohex**



Ti content	Orientation	Ti-Si region (µm)
1.75 wt%	Parallel	0.82 ± 0.20
	Perpendicular	1.60 ± 0.68
4.5 wt%	Parallel	2.16 ± 0.88
	Perpendicular	1.42 ± 0.45

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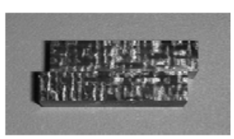
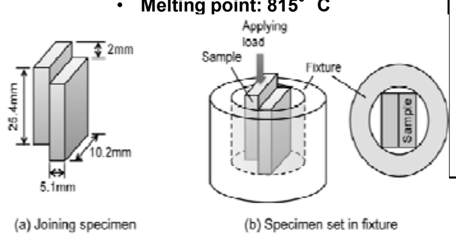
Procedure for Mechanical Evaluation

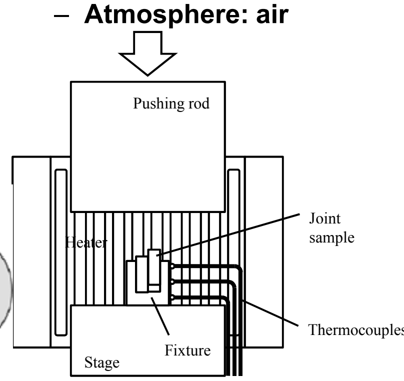
- **Joint Materials**
  - **SA-THX(⊥& ||)/Cusil-ABA**



- **Single lap offset shear test**
  - **Modified ASTM D905**
  - **R.T., 250, 650 and 750°C**
    - **Melting point: 815° C**



- **Testing Machine**
  - **Capacity: 22.5 kN**
  - **Crosshead speed: 0.5mm/min**
  - **Atmosphere: air**



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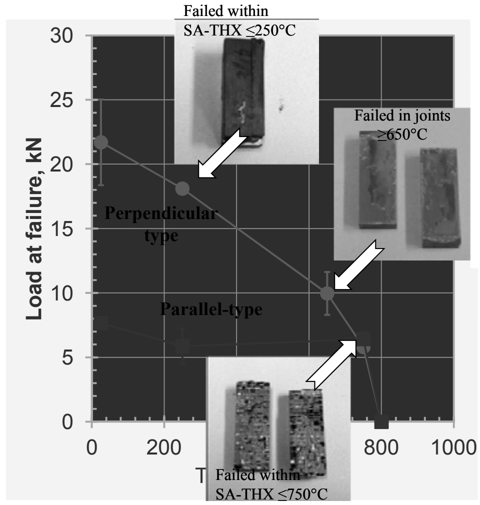
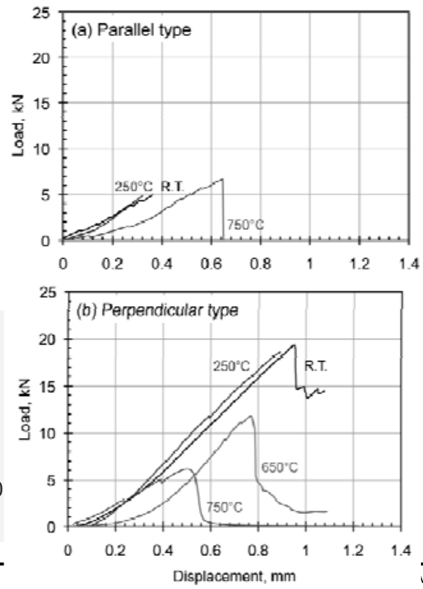
Mechanical Behavior SA-THX Joints as a Function of Test Temperature





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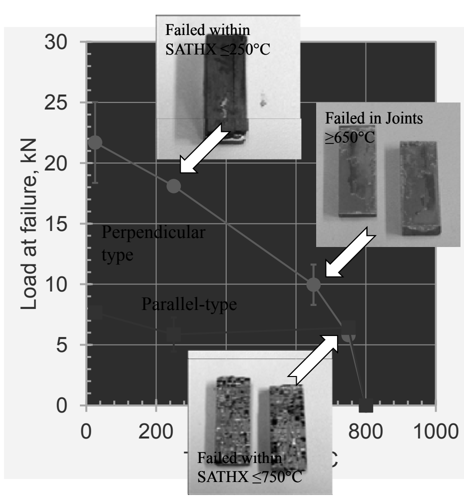
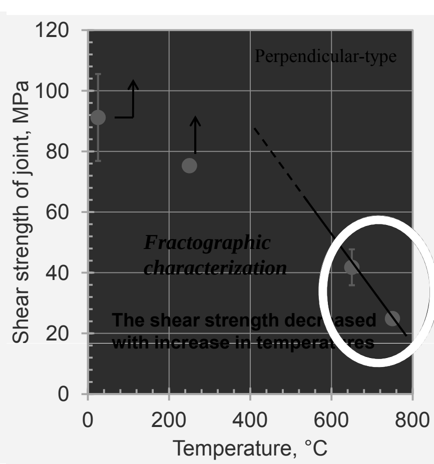
Shear Strength of the Perpendicular-Type SA-THX Joints as a function of Test Temperature





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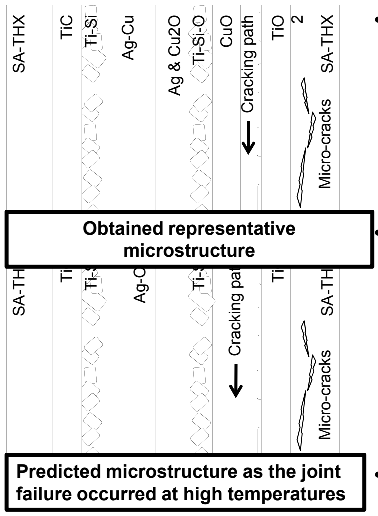
Summary of Fractography



- **Failure occurred between the filler metal and TiO_2 interaction layer**
  - TiO_2 layer may be caused by oxidation of TiC, which was formed by the reaction between Ti in the filler metal and C in SA-Tyrannohex during the brazing.
- **There are micro-cracks in SA-Tyrannohex in the vicinity of the interaction phase.**
  - The micro-cracks could be brought about by degradation of SA-Tyrannohex strength, which would be caused by C migration in SA-Tyrannohex to form TiC.
- **Since Cu reacts with O_2, CuO and Cu_2O phases were formed during the cooling after the test at 750˚C.**

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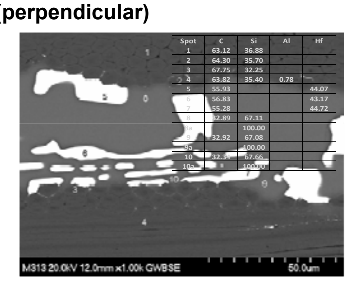
Ceramic Joining and Integration - Joining with Si-Hf Eutectic Phase Tape

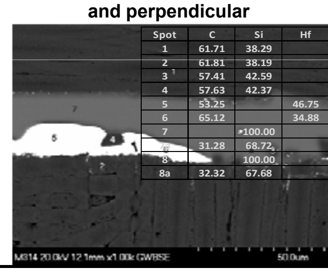
Joining with Eutectic Phase Tapes (1 layer and 2 layers)

- **SA-Tyrannohex (parallel) and SA-Tyrannohex (perpendicular)**
- **Joined with 2 mm offset for mechanical tests.**
- **Testing at R.T., 700ºC and 1200ºC**

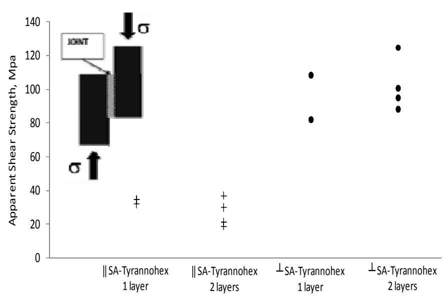


Spot	C	Si	Al	Hf
1	63.12	36.88		
2	64.30	35.70		
3	67.75	32.25		
4	63.82	35.40	0.78	
5	55.93			44.07
6	56.83			43.17
7	55.28			44.72
8	32.89	67.11		
8a		100.00		
9	32.92	67.08		
9a		100.00		
10	32.34	67.66		
10a		100.00		

SEM and EDS of joints for parallel and perpendicular



Spot	C	Si	Hf
1	61.71	38.29	
2	61.81	38.19	
3	57.41	42.59	
4	57.63	42.37	
5	53.25		46.75
6	65.12		34.88
7		100.00	
7a	31.28	68.72	
8		100.00	
8a	32.32	67.68	



Apparent Shear Strength at Room Temperature

SiC Fiber Bonded Ceramics

Long Term Challenges

Environmental Issues in Manufacturing and Product Life Cycle Management

- **ISO 14000 Standard for Environmental Management.**
- **ISO 14001 and ISO 14004 deal with Environmental Management System (EMS)- 1996.**
- **EMS provides a framework for an organization to manage the impact of its activities on the environment.**
- **Provides tools to help companies realize their own environmental policies, objectives, and targets.**
- **In Europe, European Community (EC) has established an Eco Management and Audit Scheme (EMAS) in 1997.**

In Global Economy, Consumer Demand of High Quality Products with Low or No Environmental Impact, Standards Will Play Major Role

Performance vs Cost Issue

- It is quite clear that CMC industry is in a real dilemma
  - *CMC users (customers) demand performance at cost, but cost is typically driven by market volume.*
  - *Small market volume means high cost and small number (or no) customers.*
- Users (customers) are willing to pay the COST if the CMC is truly enabling.

Concluding Remarks

- Fiber Bonded Ceramics have a lot of potential for niche high temperature applications but the manufacturing processes are still evolutionary. Their use has been limited due to limited manufacturing base and cost.

- For the wide scale applications of these materials, reliable processes and properties have to be demonstrated at various levels (coupons to full scale components). In addition, multiscale modeling tools have to be developed and effectively utilized.

- The CMC community has to leverage their resources and make a concerted effort in finding out multiple applications and educating customers. High market volume will drop the cost and will be able to sustain the supplier base.

Acknowledgments

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- Prof. Rajiv Asthana, University of Wisconsin-Stout, WI